

Gypsum Amendment and Exchangeable Calcium and Magnesium Affecting Phosphorus and Nitrogen in Runoff

N. Favaretto,* L. D. Norton, B. C. Joern, and S. M. Brouder

ABSTRACT

Loss of N and P from soil to water is not only an agronomic but also an environmental and human health problem. The main objective of this study was to evaluate the effect of gypsum amendment and soil-exchangeable Ca and Mg on the concentration and loss of nutrients in runoff, sediment, and soil. The experiment was performed in the laboratory using a programmable rainfall simulator and erosion pans. The soil was a Miami silt loam, and treatments were (i) gypsum amendment, (ii) five target exchangeable Ca/Mg ratios, and (iii) a control. Nitrogen, P, and K were applied in all treatments based on soil test results and standard recommendations for corn (*Zea mays* L.). The fertilizers were mixed to a 2.5-cm depth before pre-wetting the soil. Gypsum was applied to the surface just before the rain. Two rainfall intensities (30 and 60 mm h⁻¹) were simulated, and infiltrated water and runoff samples were taken every 5 min over 2 h. Nitrate N, NH₄-N, and P in runoff, sediment, and soil were determined. The results showed that gypsum, as compared with the control, significantly decreased the mass loss in runoff of dissolved reactive P (DRP), total P (TP), soluble NH₄-N, and total N by 85, 60, 80, and 59%, respectively. The concentration of these constituents was also significantly decreased by 83, 52, 79, and 50%, respectively. Nitrate N concentration in runoff was not decreased by gypsum. Gypsum also affected sediment and soil water-extractable P. The only significant effect of exchangeable Ca/Mg ratios was observed in the sediment water-extractable P, where Ca-treated soil decreased by 50% the concentration of P compared with Mg-treated soil. The results of this study demonstrated the potential of gypsum to decrease transport of total N and total P in runoff and the importance of Ca in the soil solution to decrease P solubility.

DECREASING P LOSSES FROM SOILS to surface waters is critical in minimizing accelerated freshwater eutrophication (Correll, 1998; Daniel et al., 1998). Similarly, the control of N in water is required not only for human and aquatic health, but also for eutrophication. Nitrogen losses from agricultural fields of the upper Midwest have been implicated as a significant contributor to the hypoxia zone in the Gulf of Mexico (USEPA, 2001). Phosphorous loss from agricultural fields is strongly associated with eroded sediment carried in runoff (Sparks, 1995; Sposito, 1989). In contrast, leaching and preferential flow are the main processes involved in the movement of N from soil to water (Smith et al., 1990). Although N and P losses to surface water can be reduced

with practices that control soil erosion and improve infiltration (Sharpley et al., 1987, 2001; Smith et al., 1990; Owens, 1994; Sharpley and Halvorson, 1994), increasing infiltration can increase nutrient losses by leaching of soluble materials.

Increasing soil aggregation and structural stability can result in increased infiltration and decreased runoff (Dontsova and Norton, 2002). Reduced infiltration and accelerated erosion rates have been observed in soils with high exchangeable magnesium (Mg) as compared with calcium (Ca) (Keren, 1989, 1991; Dontsova and Norton, 2002). This was attributed to the thinner hydration radius of Ca and its effect on clay flocculation (Sparks, 1995).

The use of mineral and by-product (synthetic) gypsum (CaSO₄ · 2H₂O) as a source of electrolytes for reclamation of sodium (Na)-afflicted soils is well known; however, its use has been recently extended to soils that have poor structure and problems with runoff and erosion. Gypsum applied at the soil surface increases the electrolyte concentration of the infiltrating rainwater, compresses the electric double layer, and provides Ca to the exchange complex where it has selectivity over Mg and Na in most soils (Shainberg et al., 1989, Dontsova and Norton, 2002). The beneficial effects of gypsum on erosion control by increasing infiltration and reducing runoff, surface sealing, and crusting have been shown in several studies (Norton and Dontsova, 1998; Norton and Zhang, 1998; Shainberg et al., 1989; Miller, 1987).

Surface and subsurface P losses are also correlated to soil P content (Sharpley et al., 2001; Pote et al., 1999). Consequently, the conversion of soil P to less soluble forms by adding materials containing Ca can reduce the potential for P pollution from soils with elevated P (Stout et al., 1998, 2000, 2003; Callahan et al., 2002). However, raising Ca concentrations in soil can affect ammonium (NH₄⁺) solubility. Koenig and Pan (1996) reported that Ca increases the concentration of NH₄⁺ in soil solution due to the preferential adsorption of Ca and the displacement of NH₄⁺ from the exchange complex.

Given that Ca and Mg behave differently in soils, that gypsum is a soluble source of Ca and electrolytes (both of which affect clay dispersion and losses of water and sediment), and that Ca can also affect the P and N solubility, the objective of this study was to evaluate the effect of a gypsum amendment and different levels of exchangeable Ca and Mg in soil on the concentration and loss of P, NH₄-N, and NO₃-N in runoff. The soil

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Abbreviations: DRP, dissolved reactive phosphorus; EC, electrical conductivity; ER, enrichment factor; I, infiltration; k_G, Gapon selectivity coefficient for Mg-Ca; NH₄-N, ammonium nitrogen; NO₃-N, nitrate nitrogen; OM, organic matter; PN, particulate nitrogen; PP, particulate phosphorus; R, runoff; SC, sediment concentration; SL, soil loss; TKN, total Kjeldahl nitrogen; TP, total phosphorus.

Table 1. Soil chemical characteristics of the original and Ca- and Mg-treated soils before receiving fertilizers.

Ca/Mg ratio		pH \parallel		K								NO ₃ -N		NH ₄ -N		P			
Target	Achieved	H ₂ O	SMP	NH ₄ OAc	NaBPh ₄	Ca	Mg	Na	CEC	OM	N total	P total	KCl	H ₂ O	KCl	H ₂ O	Bray	H ₂ O	S
						cmol _c kg ⁻¹				%				mg kg ⁻¹					
90/10	89/11 (8.0)§	5.8	6.7	0.39	1.05	8.24	1.03	0.04	18.41	2.45	0.14	0.06	10.29	8.18	6.51	1.24	44.62	3.44	46.0
80/20	83/17 (4.7)	5.8	6.7	0.44	0.97	7.74	1.63	0.05	19.41	2.35	0.15	0.06	10.07	7.97	5.89	1.26	43.77	3.41	49.0
50/50	64/36 (1.8)	5.8	6.7	0.41	1.10	6.35	3.50	0.06	16.56	2.65	0.14	0.06	11.91	9.66	6.37	1.44	43.28	3.21	43.5
20/80	41/59 (0.7)	5.9	6.7	0.44	1.03	4.00	5.83	0.04	16.89	2.40	0.15	0.06	11.60	9.39	7.29	1.43	51.55	4.01	49.0
10/90	30/70 (0.4)	5.9	6.7	0.43	1.22	2.92	6.70	0.05	16.25	2.40	0.15	0.06	10.24	8.32	7.07	1.62	52.16	4.67	47.5
Original	69/31 (2.2)	5.8	6.7	0.76	1.53	6.14	2.77	0.05	15.71	2.90	0.16	0.06	10.63	8.53	7.16	1.47	53.28	6.05	16.5
Gypsum [†]	76/24 (3.2)	5.2	—	0.72	1.50	8.59	2.67	—	—	—	—	—	55.08	50.41	45.09	21.97	54.86	2.38	—
Control [‡]	70/30 (2.4)	5.5	—	0.71	1.55	6.25	2.65	—	—	—	—	—	54.98	49.71	43.51	14.35	56.35	4.69	—

† Original soil mixed with gypsum (2.5 g of analytical-grade CaSO₄ · 2H₂O per kg soil = 5000 kg ha⁻¹) plus N (88 mg of N as NH₄NO₃ per kg soil = 176 kg N ha⁻¹). Data from Favaretto (2002).

‡ Original soil mixed with N (88 mg of N as NH₄NO₃ per kg soil = 176 kg N ha⁻¹). Data from Favaretto (2002).

§ Values in parentheses represent the amount of Ca for each Mg unit.

¶ Means of two laboratory replicates. pH water (1:1), pH SMP (Shoemaker, McLean, and Pratt buffer solution). K (1 M NH₄OAc at pH 7 and NaBPh₄). Ca, Mg, and Na (1 M NH₄OAc at pH 7). CEC = cation exchange capacity (1 M NH₄OAc at pH 7). OM = soil organic matter (1.72 × soil organic carbon). Total N and P (Kjeldahl). NO₃-N and NH₄-N (2 M KCl and H₂O). P (Bray and H₂O). S (monocalcium phosphate).

examined was from a common agricultural field of the upper Midwest, but this study was conducted under controlled conditions to allow us to compare the losses with and without the electrolyte effect and to determine the relative importance of the Ca/Mg ratio and the electrolyte effect for minimizing loss of P and N.

MATERIALS AND METHODS

This experiment consisted of seven treatments arranged in a completely randomized design with three replicates and was performed in the USDA-National Soil Erosion Research Laboratory. A programmable rainfall simulator was used on a Miami silt loam (fine-loamy, mixed, mesic Oxyaquic Hapludalf) collected from an agricultural field at the Throckmorton Purdue Agricultural Center, located south of Lafayette, Indiana. The soil surface layer (15 cm) was collected at field moisture, air-dried, and sieved by hand through a 4-mm screen.

The treatments were (i) control, (ii) surface applied gypsum (5000 kg ha⁻¹ of analytical-grade CaSO₄ · 2H₂O), and (iii) alteration to five different target exchangeable Ca/Mg ratios in soil (90/10, 80/20, 50/50, 20/80, 10/90). In this study, treatments with high (90/10 and 80/20) and low (10/90 and 20/80) exchangeable Ca/Mg ratios are also referred to as Ca-treated soil and Mg-treated soil, respectively. The gypsum rate we selected was based on the study of Agassi et al. (1982). These authors tested different rates of phosphogypsum on infiltration and runoff and found the best results using 5000 kg ha⁻¹ applied at soil surface. To change the soil-exchangeable Ca and Mg ratios,

the soil was placed in plastic boxes (88.5 cm by 42.5 cm by 33cm), slowly saturated (approximately 12 h) by capillarity action via three hoses connected to the bottom of each box, kept saturated for 1 d, and then allowed to freely drain. This procedure was repeated five times during 1 week with a 1 N solution containing the specific CaCl₂/MgCl₂ ratio required for the different Ca/Mg ratio treatments. At the end of the saturation process, the soil was leached several times (approximately three pore volumes) with a 0.005 N solution containing equal amounts of CaSO₄ and MgSO₄ to obtain an electrical conductivity (EC) similar to the original soil. The soil was then air-dried and sieved by hand through a 4-mm screen. Some soil and solution chemical characteristics of the original soil and Ca- and Mg-treated soils are presented in Tables 1 and 2, respectively. For soil chemical characterization, two soil samples of each treatment were analyzed using the following methodology: water pH (1:1 w/v soil/solution) and Shoemaker, McLean, and Pratt buffer pH (Watson and Brown, 1998); K, Ca, Mg, and Na extracted with 1 M NH₄OAc at pH 7 (Warnecke and Brown, 1998); K extracted with NaBPh₄ (sodium tetraphenylboron) modified from Cox et al. (1999); cation exchange capacity with 1 M NH₄OAc at pH 7 modified from Sumner and Miller (1996); organic carbon by chromic acid oxidation (Combs and Nathan, 1998); total Kjeldahl N (TKN) (Diamond, 2001) and total Kjeldahl P (TP) (Diamond, 2000); nitrate nitrogen (NO₃-N) and ammonium nitrogen (NH₄-N) extracted with 2 M KCl (Prokopy, 1997a; Sechtig, 1992) and with water (Wendt, 1999; Prokopy, 1997b); Bray-1 P (Bogren and Hofer, 2001) and water extractable P (Self-Davis et al., 2000; Diamond, 1995); and S by monocalcium phosphate extraction procedure (Combs et al., 1998). The soil solution was

Table 2. Soil solution characteristics of the original and Ca- and Mg-treated soils before receiving fertilizers.

Ca/Mg ratio target	K§	Ca	Mg	P	NH ₄ -N	NO ₃ -N	EC	kG (Mg-Ca)
				mg L ⁻¹			dS m ⁻¹	
90/10	12.79	125.43	14.37	0.13	1.19	2.25	0.62	0.66
80/20	12.43	121.10	23.36	0.13	1.06	3.71	0.66	0.66
50/50	14.52	83.60	44.32	0.12	1.25	10.39	0.64	0.63
20/80	11.92	39.45	63.36	0.16	1.38	4.40	0.59	0.55
10/90	13.33	26.51	70.88	0.20	1.31	2.14	0.59	0.52
Original	22.61	34.47	14.76	0.26	1.23	1.48	0.27	0.64
Gypsum†	73.70	572.00	202.40	0.09	16.86	158.80	3.09	—
Control‡	42.68	115.60	52.00	0.13	9.73	163.90	1.12	—

† Original soil mixed with gypsum (2.5 g of analytical-grade CaSO₄ · 2H₂O per kg soil = 5000 kg ha⁻¹) plus N (88 mg of N as NH₄NO₃ per kg soil = 176 kg N ha⁻¹). Data from Favaretto (2002).

‡ Original soil mixed with N (88 mg of N as NH₄NO₃ per kg soil = 176 kg N ha⁻¹). Data from Favaretto (2002).

§ Means of three laboratory replicates. Soil solution extracted by saturation paste using centrifuge instead of vacuum. EC = electrical conductivity. k_G = Gapon selectivity coefficient calculated based on binary-exchange system.

extracted from a saturated paste in triplicate (Rhoades, 1996) using a centrifuge instead of a vacuum, and EC and concentrations of K, Ca, Mg, P, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$ were determined. Calcium and Mg were determined via atomic absorption; K and Na via flame emission with an atomic absorption spectrophotometer; N and P via flow injection using a Lachat instrument; S, Zn, Mn, Cu, and Fe via inductively coupled plasma spectrometry; and B via colorimetry. Analysis of pH, organic carbon, S, and micronutrients was performed by Midwest Labs, Inc. (Omaha, NE). The other analyses were performed at the USDA-ARS-National Soil Erosion Research Laboratory. The Gapon selectivity coefficient for Mg-Ca (Sparks, 1995) was calculated as:

$$k_G = [\text{Mg} - \text{soil}] \times (\text{Ca}^{2+}) / [\text{Ca} - \text{soil}] \times (\text{Mg}^{2+})$$

where parentheses represent the solution ion activity in mol L^{-1} and brackets represent the concentration in the exchanger phase in $\text{cmol}_c \text{ kg}^{-1}$. Activities of cations in the solutions were calculated using the Davies equation, and the ionic strength was estimated using the Marion-Babcock equation (Sposito, 1989). To calculate the k_G of Mg-Ca, the system was assumed to be only a binary exchange, and K and $\text{NH}_4\text{-N}$ were ignored.

The soil was placed in an interrill erosion pan (Bradford et al., 1987) (32-cm wide, 45-cm long [0.14 m^2], and 20-cm deep) with three drainage holes (3-mm i.d.) at the bottom. The erosion pan had a 13-cm bottom layer of gravel, a 4.5-cm intermediate layer of sand, and a 2.5-cm top of soil packed at a bulk density of 1.2 g cm^{-3} to simulate the density of a normal seedbed condition. The use of a relatively small amount of soil was justified by the results obtained by Zhang et al. (1997), who demonstrated that the effective depth of the mixing zone seemed to be less than 3–4 mm. The soil was fertilized and mixed just before packing and pre-wetting. Nitrogen, P, and K were applied at the following rates (on a surface-area basis): 180 kg N ha^{-1} as NH_4NO_3 and $\text{NH}_4\text{H}_2\text{PO}_4$; 45 kg P ha^{-1} as $\text{NH}_4\text{H}_2\text{PO}_4$; and 140 kg K ha^{-1} as K_2SO_4 . The amount of N from P fertilization ($\text{NH}_4\text{H}_2\text{PO}_4$) was subtracted from the N rate to calculate the application of NH_4NO_3 . For the exchangeable Ca/Mg ratio treatments, an additional amount of K equivalent to 52 kg ha^{-1} of K as K_2SO_4 on a surface area basis was mixed into the soil to increase the exchangeable K in the Ca/Mg-treated soil to a level similar to that of the control. The amount of K to be applied to reach the exchangeable K in the control was determined by a previous study with different rates of all five exchangeable Ca/Mg ratio treatments (data not shown). Gypsum (5000 kg ha^{-1}) was applied to the surface with a 1-mm sieve just before the rain and was not incorporated.

Before rainfall simulation, the soil was pre-wetted from the bottom (3-cm tension overnight followed by 1-cm tension 1 h just before rainfall) with deionized water ($\text{EC} < 0.018 \text{ dS m}^{-1}$). A 5-cm tension at the center of the pan was applied 5 min before the rain and maintained during the rain with the slope set at 5%.

To simulate natural rain, a programmable rainfall simulator, equipped with four nozzles (80100 Veejet nozzles; Spraying Systems Inc., Weaton, IL) spaced 1.1 m apart and placed 2.4 m above the soil surface (Neibling et al., 1981), was used with deionized water ($\text{EC} < 0.018 \text{ dS m}^{-1}$). The spatial variation below the simulator operating at 6 psi was $\pm 5\%$. The rainfall rate was calibrated by adjusting the number of sweeps per minute and the water pressure. Rainfall was applied for 2 h, the first 90 min with a target rate of 30 mm h^{-1} and the last 30 min with a target rate of 60 mm h^{-1} . Two erosion pans were rained on during each simulated rainfall event. The simulated rainfall event, which had an initial period of low-intensity

rainfall followed by a period of high-intensity rainfall, was based on the results obtained by Dontsova and Norton (2002). They suggested that a low-intensity rainfall would better demonstrate the Ca effect on a soil with low structural stability, such as the Miami soil selected for this study. The rainfall rate was then increased to permit investigation of the capacity of the soil to resist the impact of the high rainfall energy. Infiltrating water and runoff plus sediment were collected every 5 min for 2 h. The sediment was separated from the runoff by drying at 105°C , and the sediment concentration was calculated by dividing the amount of sediment by the runoff plus sediment volume. The runoff and infiltration volumes were determined by weight assuming a water density of 1 kg dm^{-3} . The steady-state infiltration, runoff, soil loss, and sediment concentration rate were calculated by averaging the last four readings at the respective rainfall intensities. From the runoff-plus-sediment sample, two subsamples for analysis of nutrients were taken every 5 min. Usually the runoff-plus-sediment volume collected during the first 10 min was not enough for nutrient analysis; however, this varied among treatments and replicates. Concentrations of dissolved reactive P (DRP), ammonium N ($\text{NH}_4\text{-N}$), and nitrate N ($\text{NO}_3\text{-N}$) in runoff were obtained from a subsample filtered through a $0.45\text{-}\mu\text{m}$ filter. They were determined by flow injection analysis using a Lachat instrument (Diamond, 1995; Prokopy, 1997b; Wendt, 1999). The water samples were filtered through a $0.45\text{-}\mu\text{m}$ filter just after the collection and were frozen for subsequent analysis. From the unfiltered subsample, TP and TKN in runoff were determined by flow injection analysis according to Liao (1998) and Wendt (1997), respectively. Particulate P (PP) was obtained by subtracting DRP from TP. For soil samples, the TKN method does not quantitatively recover compounds containing N-N or N-O linkages, such as NO_3 , although it typically includes some of this N (Bremner, 1996). Therefore, it is not recommended to consider that soil TKN recovers just $\text{NH}_4\text{-N}$ and organic N. However, because the water standard samples (known concentrations of $\text{NO}_3\text{-N}$) subjected to the TKN digestion did not present any recovery of $\text{NO}_3\text{-N}$ and the concentration of soluble $\text{NO}_3\text{-N}$ obtained in runoff was low, the soluble $\text{NH}_4\text{-N}$ was subtracted from TKN to obtain particulate N (PN), which represents the N associated with sediment in runoff ($\text{NH}_4\text{-N}$ adsorbed plus organic N). Average event concentrations were calculated using all values during the 2 h of rain. Total loss of a given nutrient was expressed in kg ha^{-1} and was obtained by adding the loss values from every 5 min sampling. The 5-min losses were calculated by multiplying the runoff volume by the respective concentration. Sediment samples, collected every 5 min in the first 90 min of rain and dried at 105°C , were combined to provide five samples (first 30 min followed by every 15 min) to analyze water-soluble P, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$. These elements were extracted according to Self-Davis et al. (2000) and determined by flow injection according to Diamond (1995), Prokopy (1997b), and Wendt (1999), respectively. After every rain, a soil sample was collected from the erosion pan, and P, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$ were analyzed and determined the same way as described for sediments. Bray-1 P (Bogren and Hofer, 2001) and electrical conductivity (Rhoades, 1996) in soil after rain also were determined.

Preliminary statistical analysis indicated that some variables had to be transformed to achieve the assumption required for an ANOVA. A regression between $\log(\text{SD})$ and $\log(\text{mean})$ was performed for each variable to determine which transformation should be used (Box et al., 1978). When necessary, the data were transformed to the logarithm (base 10) of the data or of the data+1 to avoid negative numbers. The ANOVA and Tukey's test ($P < 0.05$) for mean comparison procedures were

performed using the Statistical Analysis System Release 8.0 (SAS, 1999).

RESULTS AND DISCUSSION

Steady-State Runoff, Infiltration, Soil Loss, and Sediment Concentration Rate

The application of gypsum significantly increased infiltration and decreased runoff for both rainfall intensities (Table 3). Infiltration and runoff in the gypsum treatment were 151% greater and 22% less than the control (average of both rainfall intensities), respectively. The effect of gypsum on infiltration was similar for both rainfall intensities, whereas for runoff it was more efficient at the low rainfall intensity. At a rainfall of 30 mm h⁻¹, runoff was decreased by 43% compared with the control, but at a rainfall of 60 mm h⁻¹, runoff was decreased by 12%. There was also a tendency for lower values of soil loss in the gypsum treatments when compared with the control (on average 28% lower for both rainfall intensities); however, this difference was not statistically significant (Table 3). In an experiment with Miami soil using a rainfall intensity of 64 mm h⁻¹ for 1 h, Norton and Dontsova (1998) found that fluidized bed combustion bottom ash, a gypsum-like by-product, significantly increased the steady-state infiltration rate. Yet, unlike the results obtained for this study, Norton and Dontsova (1998) observed that the steady-state soil loss rate was greater in the fluidized bed combustion bottom ash treatment than in the control.

Sediment concentration values were similar among treatments, showing that the amount of soil lost was proportional to the runoff volume at both rainfall intensities (Table 3). Raindrop splash and surface flow are the main processes involved in interrill soil erosion, with splash providing the detachment energy and flow providing the transport capacity. Most likely, gypsum applied evenly on the soil surface did not significantly affect soil detachment. This interpretation is supported by Shainberg et al. (1989), who also assumed that the rain detachment is similar in gypsum-treated and untreated soils. At the greater rainfall intensity, gypsum slightly decreased sediment concentration, although not significantly (Table 3), indicating a possible effect on soil erodibility. According to Shainberg et al. (1989), the main effect of gypsum on soil erosion is due to the reduced capacity of runoff water to cause soil loss. Gypsum

reduces runoff volume and may increase surface roughness and the tortuosity of the flow paths to reduce soil loss (Shainberg et al., 1989).

Exchangeable Ca/Mg ratio treatments did not affect runoff, infiltration, soil loss, or sediment concentration, even with the low rainfall intensity (Table 3). A slight but not significant difference (about 10%) in steady-state runoff and infiltration rate with rainfall of 30 mm h⁻¹ was observed between Ca-treated (Ca/Mg 90/10) and Mg-treated (Ca/Mg 10/90) soils. Our results are similar to Dontsova and Norton (2002). This study examined several soils using a rainfall intensity of 64 mm h⁻¹ for 1 h and also did not find differences in steady state-infiltration, runoff, or soil loss rate between Ca-treated and Mg-treated Miami soils. However, differences in infiltration and runoff were observed for two other soils (Catlin and Blount), both of which had greater aggregate stability as measured by wet sieving than the Miami soil. The authors concluded that at 64 mm h⁻¹ for 1 h, the energy of the rain was probably great enough to destroy aggregates in a soil of relatively low aggregate stability (Miami soil) irrespective of Ca/Mg treatment; they suggested that at lower rainfall intensities Ca may still have a beneficial effect.

Phosphorus Concentration and Loss in Runoff

The lowest concentration and losses of dissolved reactive P in runoff were obtained in the gypsum treatment (Table 4). Dissolved reactive P losses were 6.8 times lower in the gypsum treatment when compared with the control. These results are similar to those obtained by Brauer et al. (2005) for gypsum application to a soil with a very high soil test P content resulting from poultry manure effluent application. They also found that the addition of gypsum was more effective in lowering DRP in runoff.

In this study, soil P solubility and runoff volume were probably the factors involved in the decrease of DRP concentration and loss. Gypsum application probably reduced DRP concentration by converting the readily desorbable soil P to less readily soluble Ca-P compounds. Stout et al. (2000) evaluated the effect of coal combustion by-products and gypsum in controlling P export from soils and found that gypsum significantly reduced DRP concentration in runoff on soils under cover of grass (products applied at the surface) but not

Table 3. Steady-state infiltration (I), runoff (R), soil loss (SL), and sediment concentration (SC) rate for the treatments under 30 mm h⁻¹ of rainfall during the first 90 min followed by 60 mm h⁻¹ of rainfall during the last 30 min.

Treatment	30 mm h ⁻¹ for 90 min				60 mm h ⁻¹ for 30 min			
	I	R	SL	SC	I	R	SL	SC
	mm h ⁻¹		Mg ha ⁻¹ h ⁻¹	g L ⁻¹	mm h ⁻¹		Mg ha ⁻¹ h ⁻¹	g L ⁻¹
Ca/Mg 90/10	8.41 b†	22.11 a	1.37 a	5.27 a	7.43 b	54.01 a	6.04 a	9.85 a
Ca/Mg 80/20	6.40 b	25.58 a	1.57 a	5.38 a	6.55 b	56.17 a	7.40 a	11.40 a
Ca/Mg 50/50	7.49 b	23.96 a	1.44 a	5.15 a	6.77 b	55.84 a	6.78 a	10.55 a
Ca/Mg 20/80	8.60 b	22.72 a	1.33 a	5.04 a	7.38 b	54.34 a	6.39 a	10.34 a
Ca/Mg 10/90	7.37 b	24.38 a	1.29 a	4.51 a	6.75 b	56.32 a	6.32 a	9.76 a
Gypsum	17.28 a	14.25 b	0.92 a	5.13 a	14.77 a	48.51 b	4.66 a	8.36 a
Control	6.73 b	24.88 a	1.41 a	4.92 a	6.02 b	55.15 a	5.92 a	9.45 a

† Means followed by the same letter are not significantly different by Tukey's test ($P < 0.05$).

Table 4. Average concentration and total loss of dissolved reactive P (DRP), particulate P (PP), total P (TP), soluble $\text{NH}_4\text{-N}$, soluble $\text{NO}_3\text{-N}$, particulate N (PN), and total Kjeldahl N (TKN) in runoff for the treatments over the 2 h of rainfall.

Treatment	DRP	PP	TP	$\text{NH}_4\text{-N}$	$\text{NO}_3\text{-N}$	PN	TKN
Concentration (mg L^{-1})							
Ca/Mg 90/10	1.26 a†	3.25 ab	4.52 ab	0.95 abc	0.05 a	6.65 ab	7.60 ab
Ca/Mg 80/20	1.46 a	4.06 a	5.51 a	1.11 abc	0.10 a	7.44 a	8.55 a
Ca/Mg 50/50	1.42 a	4.30 a	5.72 a	0.90 bc	0.03 a	8.46 a	9.37 a
Ca/Mg 20/80	2.04 a	3.42 ab	5.46 a	2.06 ab	1.81 a	6.57 ab	8.41 a
Ca/Mg 10/90	1.65 a	2.88 ab	4.54 ab	1.85 ab	1.29 a	5.99 ab	7.84 a
Gypsum	0.35 b	2.31 b	2.66 b	0.55 c	0.10 a	4.15 b	4.70 b
Control	2.04 a	3.48 ab	5.51 a	2.57 a	2.28 a	6.93 ab	9.37 a
Loss (kg ha^{-1})							
Ca/Mg 90/10	0.39 a	1.79 ab	2.18 ab	0.31 bc	0.01 a	3.57 ab	3.88 ab
Ca/Mg 80/20	0.57 a	2.51 a	3.07 a	0.42 ab	0.02 a	4.63 a	5.05 a
Ca/Mg 50/50	0.46 a	2.50 a	2.96 a	0.32 bc	0.01 a	4.85 a	5.17 a
Ca/Mg 20/80	0.56 a	2.02 ab	2.58 ab	0.41 ab	0.14 a	3.82 ab	4.23 ab
Ca/Mg 10/90	0.65 a	1.98 ab	2.63 ab	0.62 ab	0.26 a	4.05 a	4.67 a
Gypsum	0.12 b	1.14 b	1.26 b	0.17 c	0.03 a	2.03 b	2.20 b
Control	0.81 a	2.32 ab	3.14 a	0.77 a	0.42 a	4.57 a	5.34 a

† Means followed by the same letter are not significantly different by Tukey's test ($P < 0.05$).

on bare soil (products mixed to 5-cm depth). According to the authors, this result indicates that in bare soil the mechanism controlling P export was detachment and transport rather than dissolution of P. In our study, total P also was dominated by particulate P, but application of gypsum at the surface on bare soil significantly reduced not only DRP but also TP. These different findings may be due to the way the gypsum was applied. Mixing gypsum into the soil diminishes its effect at the surface where most of the P transport occurs. Sharpley (1985) found values of effective depth of surface soil-erosion interaction between 0.1 and 3.7 cm, and Zhang et al. (1997) found an effective depth of less than 3 to 4 mm. Agassi et al. (1982) verified that spreading phosphogypsum on the soil surface was more beneficial for increasing infiltration and decreasing runoff than mixing to a depth of 0.5 cm.

The different exchangeable Ca/Mg ratios did not affect DRP concentration or loss in runoff (Table 4). The lack of a difference in DRP concentration among the exchangeable Ca/Mg ratio treatments and the greater effect of the gypsum treatment compared with all other treatments showed that Ca solution concentration was more important than exchangeable Ca on decreasing P solubility. Compared with the control, Ca-treated soil showed a slight tendency to reduce concentration and loss of DRP, even though it was not statistically different (Table 4).

As runoff increased, PP, which includes P adsorbed on soil particles and organic matter, increased as a function of the increased soil loss caused by flowing water (Fig. 1). Sharpley et al. (1992) found that concentration and loss of PP were reduced by practices that control erosion and runoff. In our study, gypsum showed a trend toward a reduction in soil loss (20–40%) when compared with the other treatments at both rainfall intensities, but this trend was not statistically different (Table 3). A similar tendency was observed with PP in runoff. Gypsum showed the lowest concentration and losses of PP; however, it was not significantly different from the control and from most of the exchangeable Ca/Mg ratio treatments (Table 4). The PP lost in the gypsum treatment varied from 1.6 to 2.2 times less than the PP lost in other treatments (Table 4). Particulate P concen-

trations, as opposed to DRP, increased as runoff and soil erosion increased over time. After 90 min, when the rainfall intensity was switched from 30 to 60 mm h^{-1} , increases in PP concentration were evident. The r values between PP concentration and soil loss were 0.83 and 0.80 at rainfall intensities of 30 mm h^{-1} and 60 mm h^{-1} , respectively (Fig. 1).

The gypsum treatment had the greatest PP/DRP concentration ratio in runoff (6.6), followed by the Ca-treated soils (2.5) and the Mg-treated soils and control (1.7). The PP/DRP concentration ratio in runoff is dependent on soil loss and sediment concentration. Sharpley et al. (2001) showed that an increase in erosion caused an increase in TP and PP and a decrease in DRP transported in runoff. Also, Sharpley et al. (1981) found an inverse linear relationship between DRP concentra-

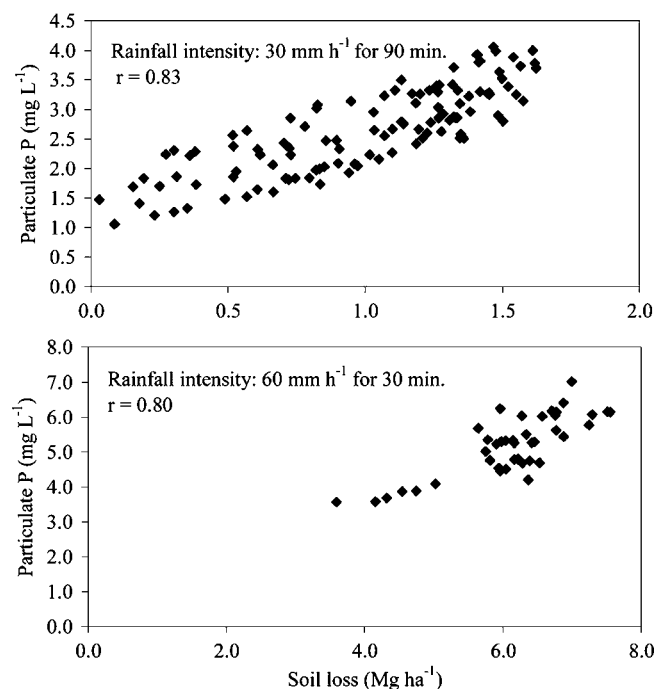


Fig. 1. Effect of soil loss on concentration of particulate P in runoff from the seven treatments during 2 h of rainfall (first 90 min under 30 mm h^{-1} followed by 60 mm h^{-1} during the last 30 min).

tion and the logarithm of sediment concentration in runoff from several cropped and grassed watersheds in the Southern Plains of the U.S. In our study, the sediment concentrations at both rainfall intensities were similar among treatments (Table 3), which is a reason to expect no differences on the PP/DRP ratio. The lower DRP concentration, which resulted in a higher PP/DRP ratio for the gypsum treatment when compared with other treatments, probably can be attributed to reduced solubility of soil P via Ca-P precipitation. Soil pH can affect Ca-P precipitation, but Stout et al. (1998) suggested that Ca may be more important than pH in reducing P solubility in the short term.

Among all treatments, approximately 75 to 91 % of TP was transported as PP, with gypsum giving the greatest percentage (Table 5). Considering that most of the total P was PP, we expected to find a similar behavior for PP and TP, and our results generally confirmed our expectations. The exception was that the gypsum treatment had significantly less TP than the control, whereas these treatments were not different for PP (Table 4). This was probably due to the high contribution of DRP to TP during the 2 h of rainfall, but especially in the first 60 min of the rainfall.

Total P losses in runoff represented a small fraction of the applied P. The smallest loss was obtained with the gypsum treatment (2.8%), whereas the greatest loss occurred in the control (7%) (Table 6). Most of the P lost was particulate P. Dissolved reactive P loss contributed a small proportion and varied from 0.26 to 1.81 % for the gypsum and control treatments, respectively.

Nitrogen Concentration and Loss in Runoff

In general, the average concentrations of $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ in runoff were low (Table 4). These N forms, especially $\text{NO}_3\text{-N}$, can easily be transported away from the runoff removal zone by infiltrated water. During the first minutes of rain, in most of the treatments and replicates, all applied water infiltrated into the soil with no surface runoff observed. The high mobility, especially for $\text{NO}_3\text{-N}$, and the low capacity of the soil to replace it probably removed most of the soluble N before the runoff started. A small variation in infiltration and runoff among replicates, or even among treatments at the beginning of the rain, caused a great difference in con-

Table 6. Percentage of the applied P lost as dissolved reactive P (DRP), particulate P (PP), and total P (TP) in runoff for the treatments in the 2 h of rainfall.

	Treatments					
	Ca/Mg 90/10	Ca/Mg 80/20	Ca/Mg 50/50	Ca/Mg 20/80	Ca/Mg 10/90	Gypsum Control
	% of the applied P lost as DRP, PP, and TP					
DRP	0.87	1.26	1.02	1.24	1.45	0.26
PP	3.98	5.58	5.56	4.49	4.40	2.53
TP	4.84	6.83	6.58	5.73	5.85	2.80

centration of N as NO_3^- or NH_4^+ . Because of the greater impact of initial infiltration on $\text{NO}_3\text{-N}$, a high variability even on the average concentration and total loss was observed. This illustrates the importance of near-surface hydraulic gradients not only on soil erosion but also on $\text{NO}_3\text{-N}$ loss.

Nitrate N average concentration and losses in runoff did not differ among treatments (Table 4). The greatest average concentration obtained was 2.3 mg L^{-1} . Over time, the runoff concentrations of $\text{NO}_3\text{-N}$ decreased, probably due to the dilution factor and losses by leaching. The highest values occurred in the first 20 min of the rainfall. Soluble N concentrations in runoff are usually low, but high values may be observed if an intense rainfall occurs just after N fertilization. However, because NO_3^- tends to leach with the initial infiltrating water, during the next storm event the soluble N runoff concentration likely will be reduced (Smith et al., 1990).

The lowest concentration and loss of soluble $\text{NH}_4\text{-N}$ in runoff was found in the gypsum treatment, although it was not significantly different from Ca-treated soils (Table 4). Gypsum amendment reduced the concentration of $\text{NH}_4\text{-N}$ in runoff 4.7-fold when compared with the control. This result likely reflects the decrease in runoff and increase in infiltration caused by gypsum addition because runoff affects the dissolution-desorption of NH_4^+ from the soil, whereas infiltration affects the leaching of NH_4^+ from the soil profile.

Increasing infiltration was expected to increase leaching of NH_4^+ and consequently to remove it from the soil surface where the runoff process occurs. Another factor that contributed to the increased transport of NH_4^+ by leaching is the solubility of NH_4^+ . Koenig and Pan (1996) found that Ca added as CaCl_2 in a silt loam soil increased $\text{NH}_4\text{-N}$ concentrations in soil solution, and they attributed this to the preferential adsorption of Ca and the displacement of NH_4^+ from soil exchange sites. In our study, the concentration of $\text{NH}_4\text{-N}$ in the soil solution extracted by saturation paste was 73% greater in the gypsum treatment when compared with the control (Table 2). Thus, if the solubility of $\text{NH}_4\text{-N}$ was greater in the gypsum treatment, then most of it could be lost by leaching, thereby reducing the concentration in runoff (Table 4).

Particulate N represents the sediment-bound N ($\text{NH}_4\text{-N}$ plus the organic N). As with PP, PN transport depends mainly on soil loss and runoff. Gypsum had the lowest PN concentration (Table 4), but it was not different from the control and from most of the exchangeable Ca/Mg ratio treatments. Loss of PN in the gypsum treatment

Table 5. Percentage of the total P (TP) as dissolved reactive P (DRP) and particulate P (PP) and of the total Kjeldahl N (TKN) as soluble ammonium N ($\text{NH}_4\text{-N}$) and particulate N (PN) for the treatments calculated using the total loss in runoff over the 2 h of rainfall.

	Treatments					
	Ca/Mg 90/10	Ca/Mg 80/20	Ca/Mg 50/50	Ca/Mg 20/80	Ca/Mg 10/90	Gypsum Control
	% of TP as DRP and PP					
DRP	18	18	16	22	25	9
PP	82	82	84	78	75	91
	% of TKN as $\text{NH}_4\text{-N}$ and PN					
$\text{NH}_4\text{-N}$	8	8	6	10	13	8
PN	92	92	94	90	87	92

was statistically different from the control (Table 4), indicating that runoff had a strong influence on controlling this parameter.

As was observed for P, concentration and losses of $\text{NH}_4\text{-N}$, $\text{NO}_3\text{-N}$, PN, and TKN did not differ among exchangeable Ca/Mg ratio treatments.

Particulate N constituted 87–94% of the total Kjeldahl N (Table 5). Sharpley et al. (1987) also observed that most of the total N in runoff occurred as PN. In a 20-watershed study, they found that on average 64% of the total N (total Kjeldahl N + $\text{NO}_3\text{-N}$) was lost as PN. In general, our results support the observation that concentration and loss of TKN in runoff (Table 4) varied with PN. However, we observed that the TKN concentration for the gypsum treatment was half that of the control but that the PN concentration was not significantly different between these two treatments. Part of the difference was probably due to the contribution of soluble $\text{NH}_4\text{-N}$ to TKN over time, but especially in the first 30 min of the rainfall.

It is expected that smaller surface loading of N occurs when runoff volumes are reduced, even though with low runoff, high concentrations can occur. However, in our experiment, mass losses of $\text{NH}_4\text{-N}$, $\text{NO}_3\text{-N}$, and TKN (Table 4) followed the same trend as concentration. As expected, the greater the runoff, the greater the load. Considering the amount of applied N, less than 1% was lost as soluble N ($\text{NH}_4\text{-N}$ + $\text{NO}_3\text{-N}$) and less than 3% as total Kjeldahl N (Table 7). Our results are in keeping with the study of Sharpley et al. (1987), who observed that 3 to 9% of the N applied to 20 watersheds in the Southern Plains of the USA was lost in surface runoff. The N losses (Table 7) represent the maximum losses because we assume that all N exported came from fertilizer with and that none came from soil N. Without considering transformations of the fertilizer N to other forms, most of the N lost in this study was associated with sediment, suggesting that practices to reduce soil loss have an environmental impact by decreasing the N load on surface waters.

Phosphorus, Nitrogen, and Electrical Conductivity in Sediment and Soil

Water-extractable P concentration in sediment (Table 8) was significantly lower in the gypsum treatment compared with the control. Water-extractable P concentrations were also affected by exchangeable Ca/Mg ratios in the Ca/Mg treatments. An exchangeable Ca/Mg ra-

Table 7. Percentage of the total applied N lost as soluble $\text{NO}_3\text{-N}$, soluble $\text{NH}_4\text{-N}$, particulate N (PN), and total Kjeldahl N (TKN) in runoff for the treatments in the 2 h of rainfall.

	Treatments						
	Ca/Mg 90/10	Ca/Mg 80/20	Ca/Mg 50/50	Ca/Mg 20/80	Ca/Mg 10/90	Gypsum	Control
% of the applied N lost as $\text{NO}_3\text{-N}$, $\text{NH}_4\text{-N}$, PN, and TKN							
$\text{NO}_3\text{-N}$	0.01	0.01	0.00	0.08	0.15	0.02	0.23
$\text{NH}_4\text{-N}$	0.17	0.24	0.18	0.23	0.34	0.10	0.42
PN	1.98	2.57	2.69	2.12	2.25	1.13	2.53
TKN	2.15	2.81	2.87	2.35	2.60	1.22	2.96

Table 8. Average concentration of sediment water extractable P, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$ over the first 90 min of rainfall at 30 mm h^{-1} .

Treatment	P _{water}	$\text{NH}_4\text{-N}_{\text{water}}$	$\text{NO}_3\text{-N}_{\text{water}}$
	mg kg^{-1}		
Ca/Mg 90/10	27.5 b†	45.9 a	2.6 a
Ca/Mg 80/20	34.3 ab	61.2 a	4.1 a
Ca/Mg 50/50	31.7 ab	59.2 a	2.8 a
Ca/Mg 20/80	44.4 ab	108.9 a	12.3 a
Ca/Mg 10/90	55.8 a	81.3 a	41.7 a
Gypsum	8.5 c	48.1 a	3.8 a
Control	48.9 a	86.3 a	40.0 a

† Means followed by the same letter are not significantly different by Tukey's test ($P < 0.05$).

tio of 90/10 had significantly less water-extractable P than an exchangeable Ca/Mg ratio of 10/90 (Table 8). This suggests that high-exchangeable Ca can decrease P solubility in sediment but not at the same magnitude as gypsum.

Water-extractable P in the soil after rain had a similar behavior as the sediment, except that there was no effect among exchangeable Ca/Mg ratios (Table 9). None of the treatments affected the content of Bray-1 P in the soil after rain. Stout et al. (1998) found that several coal combustion by-products (gypsum-like materials) had a greater effect on water-extractable P than on Bray-1 P and Mehlich-III P levels, suggesting that, in the short term, Ca may be more important than pH or liming effects in reducing P solubility in soil.

The finer-sized particles removed in the erosion process usually result in an enrichment of P in the sediment, which means that the eroded material usually has a greater P content compared with the soil (Sharpley et al., 2001). The P enrichment ratio is calculated by dividing the P content in sediment by the P content in the soil before rain. Dividing the level of sediment water-extractable P (average of 90 min) by the level of soil water extractable P after the rain (Table 9), an enrichment factor > 1 was found only for Mg-treated soils and the control. This could be due to the effect of gypsum and Ca on clay flocculation. Even with no differences in soil loss and sediment concentration, the runoff particle sizes (not measured) perhaps were different, with smaller-sized particles in the Mg-treated soil and control compared with Ca-treated soil and gypsum.

There was no effect of treatments on water-extractable $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ in the sediment (Table 8). In

Table 9. Soil Bray-1 P, water extractable P, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$, and electrical conductivity (EC) after 2 h of rainfall and enrichment ratio (ER).

Treatment	P _{Bray-1}	P _{water}	$\text{NH}_4\text{-N}_{\text{water}}$	$\text{NO}_3\text{-N}_{\text{water}}$	EC	ER‡
	mg kg^{-1}				dS m^{-1}	
Ca/Mg 90/10	131.4 a†	40.5 a	46.0 bc	1.6 bc	0.77 b	0.68
Ca/Mg 80/20	126.7 a	38.0 a	51.2 abc	6.5 ab	0.86 b	0.92
Ca/Mg 50/50	128.6 a	41.1 a	43.5 c	2.7 abc	0.59 b	0.80
Ca/Mg 20/80	128.6 a	40.4 a	43.5 c	2.4 bc	0.55 b	1.10
Ca/Mg 10/90	133.5 a	42.0 a	50.6 abc	4.5 abc	0.66 b	1.34
Gypsum	129.9 a	17.1 b	59.4 a	0.5 c	2.30 a	0.50
Control	133.6 a	40.9 a	56.8 ab	14.8 a	0.73 b	1.19

† Means followed by the same letter are not significantly different by Tukey's test ($P < 0.05$).

‡ ER = enrichment ratio obtained by dividing sediment water-extractable P by soil water-extractable P after the rainfall.

soil after rain, the gypsum treatment had the greatest water-extractable $\text{NH}_4\text{-N}$ concentrations, although the gypsum treatment value was not significantly different from that of the control (Table 9). Koenig and Pan (1996) verified that Ca significantly increased $\text{NH}_4\text{-N}$ concentrations in soil solution. The fact that the analysis of soil water-extractable $\text{NH}_4\text{-N}$ was done after 2 h of applied rainfall and resulted in no differences between control and gypsum treatments (Table 9) probably reflects the effect of leaching.

For water-extractable $\text{NO}_3\text{-N}$, an effect of gypsum on the soil was observed after rain (Table 9). Gypsum resulted in statistically less water-extractable $\text{NO}_3\text{-N}$ than for the control, but no difference in water extractable $\text{NO}_3\text{-N}$ was observed between the gypsum treatment and most exchangeable Ca/Mg ratio treatments. This probably was due to the greater infiltration rate on the gypsum treatment, leaching most of the soluble $\text{NO}_3\text{-N}$.

As expected, the electrical conductivity of the gypsum treatment in the soil after rain was much greater compared with other treatments (Table 9). The gradual dissolution of gypsum applied to the soil keeps the electrical conductivity at a level adequate to provide for clay flocculation with beneficial consequences on infiltration and runoff.

CONCLUSIONS

Gypsum applied to the soil surface as a Ca electrolyte source significantly decreased the concentration and the mass loss of DRP and TP in runoff compared with the control. The beneficial effect of gypsum also was observed for soluble $\text{NH}_4\text{-N}$ and total Kjeldahl N in runoff; however, there was no effect of gypsum on $\text{NO}_3\text{-N}$ in runoff. Gypsum reduced the water-extractable sediment P level but did not alter the sediment water-extractable $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$. In general, exchangeable Ca/Mg ratios did not affect concentrations or losses of N and P in any of the analyzed forms. The exception was for sediment water-extractable P, where the high exchangeable Ca treatment decreased P concentration compared with the Mg-treated soil.

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